

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
3 October 2002 (03.10.2002)

PCT

(10) International Publication Number
WO 02/077613 A2

(51) International Patent Classification: **G01N 9/00**,
11/16, B81B 3/00, G01N 29/02, E21B 47/00, G01N 33/28

Panama City (PA). **SCHLUMBERGER HOLDINGS LIMITED** [—/—]; P.O. Box 71, Craigmuir Chambers, Road Town, Tortola (VG).

(21) International Application Number: PCT/US02/09025

(22) International Filing Date: 22 March 2002 (22.03.2002)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
60/278,302 23 March 2001 (23.03.2001) US

(72) Inventors; and

(75) Inventors/Applicants (*for US only*): **GOODWIN**, Anthony [US/US]; 599 High Street Extension, Thomaston, CT 06787-1212 (US). **DONZIER**, Eric [FR/US]; 146 Candlewood Lake Road, Brookfield, CT 06804 (US). **MANRIQUE**, Maria [ES/GB]; 61 College Fields, Woodhead Drive, Cambridge CB4 1YZ (GB). **PELHAM**, Sarah [GB/GB]; 5 Napier Street, Cambridge CB1 1HR (GB). **MEETEN**, Gerry [GB/GB]; South Cottage, Bromley Lane, Stanton, Ware, Herts SG11 1NW (GB).

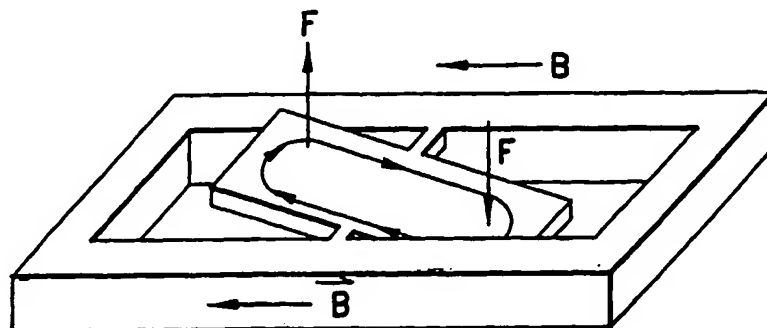
(71) Applicants (*for all designated States except US*): **SERVICES PETROLIERS SCHLUMBERGER** [FR/FR]; 42, rue Saint-Dominique, F-75007 Paris (FR). **SCHLUMBERGER CANADA LIMITED** [CA/CA]; 525 - 3rd Avenue S.W., Calgary, Alberta T2P 0G4 (CA). **PRAD RESEARCH AND DEVELOPMENT N.V.** [NL/NL]; De Ruyterkade 62, Willemstad, Curacao (AN). **SCHLUMBERGER TECHNOLOGY B.V.** [NL/NL]; Parkstraat 83-89, NL-2514 JG The Hague (NL). **SCHLUMBERGER OVERSEAS S.A.** [PA/PA]; 8 Calle Aquillo de la Guardia, Panama City (PA). **SCHLUMBERGER SURENCO S.A.** [PA/PA]; 8 Calle Aquilino de la Guardia,

(74) Agents: **BATZER**, William, B. et al.; Schlumberger-Doll Research, Intellectual Property Law Department, Old Quarry Road, Ridgefield, CT 06877-4108 (US).

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW.

[Continued on next page]

(54) Title: FLUID PROPERTY SENSORS



(57) Abstract: Micro-Electro Mechanical Systems (MEMS) based fluid sensors adapted to measure physical properties of oilfield reservoir fluids under downhole conditions. Certain embodiments of the invention may be characterized as a MEMS based reservoir fluid sensor adapted for downhole conditions having a planar member machined from a substrate material, an electrical conductor formed at least partly on the planar member; and a gauge formed on the planar member and adapted to measure a physical effect on the planar member, the physical effect being indicative of a property of a fluid in contact with the planar member.

BEST AVAILABLE COPY

Published:

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

FLUID PROPERTY SENSORS

This patent application claims priority from U.S. Provisional Patent Application No. 60/278,302, filed on March 23, 2001, which is incorporated herein by reference.

5

FIELD OF THE INVENTION

This invention relates to fluid property sensors and, more particularly, to sensors that are capable of measuring thermophysical (thermodynamic and transport) properties of fluids, such as oilfield fluids, under difficult conditions, such as downhole.

10

BACKGROUND

In the oil exploration and production industries, it is important to know the density and viscosity of reservoir fluids, such as crude oil or brine, at the pressure and temperature of the reservoir. These properties are used to determine the permeability and flow characteristics of the reservoir as well as the commercial value of the fluid in place. It is current practice to obtain a fluid sample downhole during the exploration phase of oilfield development and to determine the fluid's thermo-physical properties at the surface. Although the pressure and temperature of the fluid sample at the surface can be adjusted to those at reservoir conditions, there is considerable difficulty in obtaining a sample that closely resembles the downhole fluid chemical composition owing to the volatility of lighter hydrocarbons, solids deposition, and drilling fluid contamination. The cost of retrieving a downhole sample and the difficulty of handling samples at the surface under downhole pressure and temperature conditions are both very high. Making measurements downhole will accelerate the evaluation process and reduce total costs. Decisions concerning reservoir production and optimization activities are often based on analyses of extremely small fluid samples obtained

25

downhole, by volume relatively less than 10^{-9} of the reserves within a typical reservoir.

The composition of a reservoir fluid can and will change during the lifetime of a reserve and thus the fluid properties will change. More frequent fluid property measurements, taken throughout the exploration and production process, would be

5 extremely useful.

Downhole conditions are far more extreme than typical fluid property sensors are capable of operating under. At this time, fluids in a majority of producing hydrocarbon reservoirs are at downhole temperatures between (50 and 175) degrees Celsius, at downhole pressures between (100 and 2,000) bar, have densities in the range
10 (500 to 1300) kg m^{-3} , and have viscosities on the order of (1 to 1000) mPa s. Oilfield equipment typically must pass rigorous shock and corrosion resistance standards, due to the difficult deployment environment and the possible presence of corrosive fluid constituents such as H_2S and CO_2 . Reservoir fluids are often extremely complex and may contain chemical components ranging from asphaltenes and waxes to methane. In
15 this environment, it is necessary to have a fluid sensor that can operate accurately in this complicated and harsh environment. No commercially available device exists today that would satisfy these requirements. Fluid property sensors are also utilized in many other industries, such as the water industry, the chemical processing industry, and the food processing industry. Improved fluid property sensors could offer substantial
20 benefits to these types of industries as well.

SUMMARY OF INVENTION

The present invention includes a range of devices, based on Micro-Electro Mechanical Systems (MEMS) technology, for analyzing or measuring thermophysical properties (such as density and viscosity) of fluids (such as oilfield reservoir fluids) under difficult conditions, such as downhole. The fluid property sensors of this invention may be embedded in a well completion or in the formation; alternatively, these devices may be incorporated into downhole sampling tools, such as the Schlumberger Modular Formation Dynamics Tester (MDT), or in sample bottles designed to hold reservoir fluid samples under downhole conditions.

In accordance with the invention, a MEMS based fluid sensor includes a planar member machined from a substrate material, an electrical conductor formed at least partly on the planar member, and a gauge formed on the planar member. The gauge is adapted to measure a physical effect on the planar member that is indicative of a property of a fluid in contact with the planar member.

In a preferred embodiment, the sensor includes: a monolithic structure, machined from a substrate material, having a support portion allowing the monolithic structure to be attached to another structure, a plate portion capable of oscillating, and a flexible beam portion that decouples stress in the support portion from motion induced stress in the plate; means for producing a magnetic field; an electrical conductor, formed at least partly on the plate, allowing current flowing through the electrical conductor to interact with the magnetic field to induce oscillation of the plate; a strain sensor, formed on the monolithic structure, adapted to detect movement of the plate; and means for determining a thermodynamic property of a fluid in contact with the plate using the strain sensor detected plate movements.

Further details and features of the invention will become more readily apparent from the detailed description that follows.

BRIEF DESCRIPTION OF FIGURES

5 The invention will be described in more detail below in conjunction with the following Figures, in which:

Figure 1 schematically illustrates a magnetic field B interacting with a current i flowing atop a planar member to generate a force F on the member in accordance with certain embodiments of the invention;

10 Figure 2 schematically illustrates one embodiment of a MEMS fluid sensor according to the invention;

Figure 3 schematically illustrates a second embodiment of a MEMS fluid sensor according to the invention;

15 Figure 4 schematically illustrates a third embodiment of a MEMS fluid sensor according to the invention;

Figure 5 schematically illustrates a fourth embodiment of a MEMS fluid sensor according to the invention;

Figure 6 schematically illustrates a fifth embodiment of a MEMS fluid sensor according to the invention;

20 Figure 7 schematically illustrates a sixth embodiment of a MEMS fluid sensor according to the invention;

Figure 8 shows one embodiment of a MEMS-fluid sensor and associated mounting hardware;

Figure 9 schematically illustrates uses of MEMS fluid sensors according to the invention in connection with certain hydrocarbon exploration and production activities;

Figure 10 shows values obtained from equation (4) plotted as a function of the resonance frequency, normalized to the resonance frequency in a vacuum ($p=0$), of a flexural plate embodiment of a MEMS-fluid sensor of the type shown in Figure 2;

Figure 11 shows fractional differences of density determined using a flexural plate embodiment of a MEMS-fluid sensor of the type shown in Figure 2 and the true density calculated from an equation of state;

Figure 12 plots quality factor as a function of $(\eta\rho)^{-1/2}$, where η is the viscosity and ρ is the density, of a flexural plate embodiment of a MEMS-fluid sensor of the type shown in Figure 2;

Figure 13 shows relative difference in viscosity (determined by subtracting the true viscosity calculated from an equation of state from the viscosity determined using a flexural plate embodiment of a MEMS-fluid sensor of the type shown in Figure 2 and then dividing the result by the true viscosity) as a function of viscosity η ; and

Figure 14 shows the frequency f versus density ρ response of a MEMS-fluid sensor of the type shown in Figure 2 over a wide range of fluid densities.

DETAILED DESCRIPTION

The present invention describes a range of electro-mechanical sensors that have been adapted for analyzing and measuring thermo-physical properties of oilfield fluids under downhole conditions. The electro-mechanical sensors of this invention are micro-machined out of a substrate material and are fabricated using technologies that have been developed to produce electronic integrated circuit (IC) devices at low cost

and in large quantities (batch fabrication). Devices of this type are typically referred to as Micro-Electro-Mechanical Systems (MEMS) devices, and the inventors believe the present invention describes the first application of MEMS technology to downhole oilfield fluid property sensing.

5 The MEMS based sensors of the present invention include a planar member machined from a substrate material, an electrical conductor formed at least partly on the planar member, and some sort of gauge formed on the planar member. When the planar member is put into contact with a fluid, such as an oilfield fluid, and the MEMS based sensor is activated, the gauge measures a physical effect on the planar member
10 that is indicative of an oilfield fluid property.

 In some embodiments of the invention, the planar member includes a movable element that is attached to the planar member, and the electrical conductor forms a conducting coil on the planar member through which electrical current (\vec{i}) flows when the sensor is activated. In the presence of a magnetic field (\vec{B}) applied approximately
15 parallel to the surface of the planar member, the flowing current interacts with the magnetic field to produce Amperian forces (\vec{F}) (also referred to as Laplace or Lorentz forces) on the movable element, as illustrated in Figure 1. The forces act on the movable element at a frequency determined by the applied current (\vec{i}) and set it vibrating or oscillating. The gauge in these embodiments senses the movement of the
20 oscillating element using a strain sensor, such as a Wheatstone bridge of piezoresistors, a back-EMF sensor, or an electrical impedance sensor. It is often advantageous to include gauges at different positions on the planar member to distinguish between different modes of oscillation. Each mode of oscillation has a characteristic resonant frequency (f^{ac}) at which the amplitude of oscillation (S) is a maximum. When these

sensor embodiments are contacted with a fluid, the oscillating element causes the fluid around it to move, adding effective mass, or inertia, to the element's intrinsic mass and causing a decrease in the resonant frequency ($f < f^{vac}$). Fluid shear around the element leads to viscous energy dissipation and causes the quality factor of the oscillator (Q^{vac}) to decrease as well ($Q < Q^{vac}$). Calibration of these sensor embodiments typically involves identifying different modes of oscillation, their resonant frequencies, and the quality factor of the oscillator.

These sensor embodiments relate measurements of the resonant frequency, f_0 , and quality factor, Q , to the density and viscosity of the fluid. For typical oilfield fluids, which have a density (ρ) in the range (500 to 1300) kg m^{-3} and viscosity (η) on the order of (1 to 1000) mPa s, the quality factor may be expressed as:

$$Q = ak \sqrt{\frac{\omega_0}{\eta \rho}} \quad (1).$$

where a is a term describing the effective mass or inertia of the oscillating element and k is a constant related to the geometry of the oscillating element. The resonant circular frequency ($\omega_0 = 2\pi f_0$) of the oscillating element may be generally expressed as

$$\omega_0 = \sqrt{c/a} \quad (2).$$

where c is a term describing the elastic restoring force or torque which acts on the element's mass to return it to its initial position following a perturbation, such as \vec{F} acting on the element. Solving equation (2) for a and substituting in equation (1) gives

$$Q = \frac{ck}{\omega_0^{3/2} \sqrt{\eta \rho}} \quad (3).$$

The constants c and k typically depend only on the geometry and composition of the oscillating element, and so may be determined by calibrating the sensor using fluids of

known density and viscosity. Moreover, assuming the Reynolds number is much greater than one, which is generally a sound assumption with these sensor embodiments and typical oilfield fluids, then the mass that the fluid movement effectively adds to the oscillating element is proportional only to the fluid density. Thus, from equation (2) it

5 follows that:

$$\frac{\omega_0^{\text{vac}}}{\omega_0} \approx (1 + k\rho)^{1/2} \quad (4).$$

Thus, fluid density (ρ) may be obtained using equation (4) by measuring ω_0^{vac} and ω_0 , and, once ρ is known, fluid viscosity (η) may be obtained from equation (3) by additionally measuring Q .

10 The density and viscosity may also be determined using the following formulas:

$$\rho(B, T, p) = \frac{4A_1}{\pi Y}.$$

$$\left\{ \frac{W\rho(Si, T, p)}{\{f(B, T, p)/f(p=0)\}^2} - W\rho(Si, T, p) - \left[\frac{A_2 \sqrt{\eta(B, T, p)\rho(B, T, p)}}{\sqrt{\pi f(B, T, p)}} \right] \right\} \quad (5).$$

and

$$15 \quad \eta(B, T, p) = \left[\frac{A_3}{Q(2\pi f(B, T, p))^{3/2}} \right]^2 / \rho(B, T, p) \quad (6).$$

where A_1 , A_2 , and A_3 are adjustable parameters that are determined by calibrating the sensor with known fluids, $\rho(B, T, p)$ is the density of the fluid being sensed (B) at temperature T and pressure p , Y is the width of the oscillating element, W is the thickness of the oscillating element, $\rho(Si, T, p)$ is the density of silicon (the

20

material the oscillating element comprises) at temperature T and pressure p , $f(B, T, p)$ is the resonant frequency of the sensor when immersed in the fluid being sensed at temperature T and pressure p , $f(p=0)$ is the resonant frequency of the sensor in a vacuum, $\eta(B, T, p)$ is the viscosity of the fluid being sensed (B) at temperature T and pressure p , and Q is the resonance quality factor that is described above.

Figures 2-4 illustrate sensor embodiments that operate according to these principles. Figure 2 schematically illustrates a flexural plate embodiment of a MEMS based sensor 20 that has a monolithic structure, planar member 24, machined from a semiconductor substrate. A portion, flexural plate 22, is thinned and cut (or etched) from the (thicker) planar member 24, and remains attached to the planar member along one side 23. An electrical conductor extends from the planar member to form a conducting coil 26 atop the plate 22. In this particular embodiment, two strain gauges 27a, 27b are shown near the corners of the side at which the plate 22 attaches to the planar member 24, where maximum strain occurs during the oscillatory movement of the plate. Computer modeling may be used to determine where the maximum strain will occur. Two strain gauges are used to allow the oscillation mode to be identified. Electrical connectors 28 are shown extending to connection pads 29 on a support portion of the structure that may be used to wire bond the sensor to a printed circuit board or otherwise connect the sensor with various electrical devices that would be used to activate and interrogate the various sensor elements. Connection pads 29 are formed on a support portion of the planar member 24 that allows the sensor 20 to be mounted or attached to another structure. Between the support portion and the plate portion of the planar member 24 is a flexible beam portion that decouples stress in the support portion from motion induced stress in the plate.

A fluid in contact with this sensor embodiment would surround the flexural plate 24 and fill the area 21 so that, when activated, the flexural plate would vibrate and cause the fluid to move and effect the resonant frequency and quality factor of the plate, as described above. Although the designs shown in Figure 2 and in subsequent Figures 5 have predominantly rectangular features, may leave a portion of the planar member 26 surrounding the plate 22, or may produce a section of the planar member 26 between the plate 22 and the connection pads 29 that is narrower in width than the plate 22, it should be recognized that these features (as well as certain other common features) represent optional, not required, features of the inventive sensors.

10 Figure 3 shows a torsional plate embodiment of a MEMS based sensor 30. A torsional plate 32 is etched or cut from a planar member 34 and remains attached to the planar member by two arms 33a-b extending from opposite sides of the plate. An electrical conductor extends from the planar member 34 and across one of the arms 33b to form a conducting coil 36 atop the plate 32. A strain gauge 37 is partially formed on 15 at least one of the arms 33a, where the maximum strain occurs during the oscillatory movement of the plate. Electrical connectors 38 may extend to connection pads (not shown) used to wire bond the sensor to a printed circuit board, or otherwise lead to connections between the sensor and various electrical devices that would be used to activate and interrogate the various sensor elements. A fluid in contact with this sensor 20 embodiment would surround the plate 32 and fill the areas 31a-b so that, when activated, the torsional plate would oscillate and cause the fluid to move and effect the resonant frequency and quality factor of the plate, as described above.

Figure 4 shows still another embodiment of a MEMS based sensor 40. This device could be used to measure viscosity. A movable plate 42 is cut or etched from a

planar member **44** and remains attached to the planar member by a plurality of arms **43a-h** extending from opposite sides of the plate. An electrical conductor extends from the planar member across some of the arms **43c-d** and **43g-h** to form a conducting coil **46** partially atop the plate **42**. A plurality of strain gauge resistors **47a-d** in the form of
5 a Wheatstone bridge is formed on some of the arms **43a-d**. Electrical connectors **48** may extend to connection pads (not shown) used to wire bond the sensor to a printed circuit board, or otherwise lead to connections between the sensor and various electrical devices that would be used to activate and interrogate the various sensor elements. A fluid in contact with this sensor embodiment would surround the plate **42** and fill the
10 areas **41a-b** so that, when activated, the movable plate would vibrate and cause the fluid to move and change the resonant frequency and quality factor of the plate, as described above. In this embodiment, a magnetic field is applied perpendicular to the plate **42** to induce shear mode oscillation of the plate.

The embodiments shown in Figures 2 to 4 take advantage of the Amperian
15 forces generated by the interaction of a current flowing in a conducting coil and a magnetic field to activate a moveable element and consequently measure the density and/or viscosity of a fluid. Figure 5 shows one embodiment of a MEMS based sensor that takes advantage of the Amperian forces acting on a moving element to acoustically induce and detect the onset of bubble formation. Knowledge of the bubble point is
20 important in oil production because if the borehole pressure drops below the bubble point pressure, gas bubbles can form in the reservoir, leading to a decrease in the oil phase relative permeability. A movable element, shown in Figure 5 as a flexural plate **52**, is etched or cut from a planar member **54**. An electrical conductor forms a conducting coil **56** atop the element **52**. In the presence of a magnetic field, a current

flowing through the conducting coil creates forces on the element that cause the element to vibrate and to act as an acoustic transducer. The acoustic waves generated by the vibrating element can, in a process known as cavitation, lead to bubble evolution in a fluid at or near its bubble point pressure and temperature. To sense the presence of bubbles, the gauge of this embodiment may include a differential pressure gauge 57a, as well as a thermometer 57b and an absolute pressure gauge 57c, as shown in Figure 5. Alternatively, changes in electrical properties of the acoustic transducer may be used to detect bubble formation, as described in U.S. Patent No. 6,128,949.

The present invention also encompasses MEMS based sensors that do not include a movable element nor rely on Amperian forces to work. For example, Figure 6 shows one embodiment of a MEMS based hot wire anemometer, which may be used to measure the thermal conductivity of a reservoir fluid. An elongated element 62 is etched or cut from a planar member 64 along two opposing sides 63a-b, so that the elongated element remains attached to the planar member along its ends 63c-d. A heater 65, such as a resistance heater, is formed atop the element 62 near one of the ends 63c, and a heat gauge 67, such as a resistance thermometer, is formed near the opposite end 63d of the elongated element from the heating element. Electrical conductors 66 couple the heater 65 and the heat gauge 67 with various electrical devices (not shown) that would be used to operate them. When in contact with a reservoir fluid and activated, the heater heats the fluid surrounding it around one end of the elongated element, and the heat gauge monitors the change in temperature as the fluid conveys the heat outwardly, including towards the opposite end of the elongated element. The time-rate of change in temperature monitored by the heat gauge may be calibrated to the fluid's thermal conductivity and viscosity.

Another example of a MEMS based sensor encompassed by the present invention is shown in Figure 7. Figure 7 shows one embodiment of a MEMS based hot wire flow calorimeter, which may be used to determine the heat capacity/thermal conductivity and phase transition temperatures of a reservoir fluid. Four heating resistors 77a-d are formed in a Wheatstone bridge atop a planar member 74. The center of the planar member where the two central resistors 77b, c are placed is etched or cut away so that fluid may flow around and between the two central resistors ensuring good thermal contact with them. The outer two resistors 77a, d do not have the same degree of thermal contact with the fluid due to the protection provided by the relatively thick planar member. When activated, the central resistors will reach a temperature different from that of the outer resistors, the difference of which depends on the heat capacity of the surrounding fluid. Due to the excellent temperature sensitivity of silicon, the temperature difference will induce an early measurable voltage imbalance in the bridge.

The heat generated by the central resistors may also be used to induce a phase transition in the surrounding fluid and so monitor the phase transition temperature. The dielectric constant of a reservoir fluid may be measured using a MEMS based sensor similar to that described above, with capacitors replacing the resistors shown in Figure 7.

Various MEMS based sensors of the types described above may be fabricated onto a single monolithic substrate or may be physically packaged together and used to measure sufficient properties to determine equation of state parameters that can then be used to calculate the physical properties of the reservoir fluids under virtually any conditions likely to be found in the reservoir fluid production stream. A different

minimum set of sensor types may be desired for each major type of fluid regime. This type of integrated sensor packaging and joint fluid property inversion is particularly helpful with respect to downhole hydrocarbon samples because adjacent sensors would be making measurements on virtually identical fluid samples and can be expected to deliver a self-consistent set of data values. Getting self-consistent data is often problematic when hydrocarbon samples being kept at downhole pressure and temperature conditions are analyzed at the surface because a series of different analysis equipment is often used and it is difficult or impossible to ensure that the fluid samples seen by each of these pieces of equipment is virtually identical.

The sensors of the present invention may be micro-machined out of a wafer substrate. Silicon, such as poly-silicon and in particular mono-crystalline silicon, with its good elastic properties and small internal vibration losses, is the presently preferred material for many embodiments of the present invention. Most integrated circuit (IC) technology has been developed for silicon processing, which is an additional advantage for the present invention. Other materials, such as silicon carbide, however, possess properties, in particular chemical stability, that may be advantageous for oilfield fluid property sensors in general, and IC-processing techniques for these materials are also known. Thus, it is to be understood that the sensors of the present invention may be made out of a variety of materials.

Embodiments of the present invention, such as those described above with reference to Figures 2 to 7, have been processed from a silicon on insulator (SOI) wafer with a 350 μ m thick silicon substrate layer and a 20 μ m thick layer of mono-crystalline silicon separated by a 0.5 μ m thick silicon oxide layer. The exposed surface of the 20 μ m thick layer of mono-crystalline silicon was oxidized to a depth of about 0.3nm

and atop this a 400nm thick layer of polysilicon was deposited. The whole assembly was annealed. The polysilicon layer was then doped with boron, coated with an UV sensitive photoresist and exposed to ultraviolet radiation in the presence of a mask. The exposed polysilicon was removed in a reactive ion etching step to the 0.3nm

5 silicon oxide layer formed at the surface of the 20 μ m thick mono-crystalline silicon layer to form piezoresistors, which are the active element of a Wheatstone bridge strain gauge. Silicon nitride (Si_xN_y) was deposited atop the surface coated in etched polysilicon, with a chemical vapor deposition step to form a chemically inert and insulating layer. The resulting layer underwent photolithography and etching steps to allow

10 electrical contact to be made to the underlying poly-silicon layer. A layer of aluminum was deposited to form the electrical contacts, and underwent photolithography and etching steps to leave a connection to the piezoresistors for wire-bonding, to form the conducting coil atop the movable element, and to provide the connection between the coil and the connection pads. A second layer of silicon nitride (Si_xN_y) was deposited

15 atop the aluminum and etched poly-silicon layers in a plasma-enhanced chemical vapor deposition step to form a chemically inert layer, which was then etched to provide an open area for wire bonding. The geometry of the movable element is defined by structuring the 20 μ m silicon layer using photolithography and plasma etching. Other types of passivation layers may be used, such as silicon carbide, diamond-like carbon,

20 and/or polytetrafluoroethylene materials. Finally, the 350 μ m mono-crystalline silicon substrate below and around the defined geometry is removed with a back-etching process to relieve the movable element.

It is to be understood that while the inventors have chosen these particular parameters (materials, dimensions, doping levels, etc.) and have used these particular

steps, other parameters and other processing steps may be used to manufacture sensors according to the present invention. Thus, the present invention is not intended to be limited to sensors manufactured according to the set of parameters or series of processing steps described herein.

5 Figure 8 shows one embodiment of a MEMS based sensor adapted to be introduced and fit to a pressure vessel, such as a reservoir fluid sample bottle of the kind commonly used to transport and store reservoir fluid samples under downhole conditions. An example of such a sample bottle may be found in commonly-owned published UK Patent Application No. GB 2,348,222 A. The MEMS based sensor
10 device **81** is shown wire-bonded to a printed circuit board **82** that is fed-through and mounted in a tube **83**. The tube **83** is fit with a compression or other type of fitting **84** capable of forming a pressure-tight seal when the assembly **80** is fit to a pressure vessel. When fit to a pressure vessel, connection pads **85** formed on the printed circuit board remain exposed to be coupled with the electrical equipment used to activate and
15 monitor the MEMS based sensor. The embodiment of Figure 8 is shown with a removable cap **86** that can be placed over the MEMS based device to provide a degree of protection from the environment while allowing fluid to contact the MEMS based device. In some embodiments, a permanent magnet (not shown) may be housed in the cap or deposited on the MEMS based device to provide a magnetic field required for
20 actuation of the device. Alternatively, an electro-magnet may be housed in the cap, or formed on the MEMS based device, for the same purpose.

A MEMS based device according to the present invention of course may be packaged differently, the assembly being adapted to use already existing electrical feed-throughs and ports on a sample bottle. In addition to measuring properties of oilfield

fluids being stored and/or transported in sample bottles, the MEMS based sensor devices may be adapted for use in downhole fluid sampling tools, such as Schlumberger's MDT tool, or in permanent reservoir monitoring applications.

Figure 9 schematically illustrates certain uses of MEMS fluid sensors according to the invention in connection with hydrocarbon exploration and production activities. In Figure 9, a fluid sampling tool 90 has been lowered within borehole 91 on a cable 92 until the probe portion 93 of the tool is adjacent to the desired formation 94. The electronics and processing unit 95 then instructs the fluid sampling tool 90 to withdraw a sample of the reservoir fluid from the desired formation 94. The withdrawn fluid sample may pass by a MEMS based sensor 96, where various fluid properties are determined, and/or may be stored within an intelligent sample bottle 97 that has an integrated MEMS based sensor that allows the fluid properties to be determined after the sample is returned to the surface.

A flexural plate MEMS based sensor embodiment, of the type shown in Figure 2, was mounted as described above and tested in fluids with densities from approximately 1 kg/m^3 to 1900 kg/m^3 and viscosities from approximately $18 \text{ } \mu\text{Pa}\cdot\text{s}$ to $2 \text{ Pa}\cdot\text{s}$, where the resonance frequency ranged from about 5000 Hz to 500 Hz and the quality factor ranged from about 200 to 2. The complex resonance frequency was determined while the device was surrounded by argon, methane, and nitrogen at densities below 145 kg/m^3 . The values obtained with argon were used to obtain the constant k in equation (4) above. The results are shown in Figure 10, as a function of the frequency normalized to f_0^{vac} . The densities obtained from the resonance frequency using equation (4) are shown in Figure 11 as fractional deviations from values obtained from the equations of state package known under the acronym NIST 14. The majority

of the differences are less than 0.5%. Similar results were obtained at densities up to 1100 kg/m³. Equation (3) shows the product of the resonance quality factor and the term $(\eta\rho)^{-1/2}$ is a constant; Figure 12 confirms this expectation, where the solid line, which fits the imaginary component of the resonance for nitrogen, represents the results for all three gases. The relative deviation of the viscosity obtained with the calibration based solely on nitrogen from that obtained from NIST 14 is, as shown in Figure 13, less than 10%. The frequency f versus density ρ response of a MEMS-fluid sensor of the type shown in Figure 2 over an extremely wide range of fluid densities, from nearly zero to 700 mg/m³, is shown in Figure 14.

These types of sensors have the significant advantage that they do not have to be calibrated for each range of fluid types the sensor is expected to encounter and for each set of operating conditions the sensor will be used in. This wide spectrum of valid operating conditions is particularly important downhole because such a broad range of downhole fluids are commonly encountered. By having a very thin plate, the sensor is very sensitive to the fluid properties. For one moving plate embodiment of the present invention, the plate is approximately 2.8 millimeters wide, approximately 2.3 millimeters long, and approximately 20 micrometers thick, resulting in the weight of the moving plate being on the order of 0.30 milligrams. In comparison, the mass of water moved by the plate is estimated to be on the same order as the mass of the plate, and these roughly similar masses helps to account for the excellent sensitivity of the device. The primary oscillation mode resonance frequency for this embodiment in a vacuum is approximately 5.3 kHz. Highly decoupled excitation and detection systems greatly simplify the electronics and ensures a very accurate measurement down to the very low values of Q that may be encountered in very viscous fluids. MEMS based

sensor embodiments of the type shown in Figure 2 are typically quite insensitive to temperature changes as well. Silicon construction aids in long term stability, as well, because silicon does not creep like many other materials.

The invention has been described herein with reference to certain examples and
5 embodiments. It will, however, be evident that various modifications and changes may
be made to the embodiments described above without departing from the scope of the
invention as set forth in the claims.

We claim:

1. A Micro-Electro Mechanical Systems (MEMS) based reservoir fluid sensor adapted for downhole conditions comprising:
 - a) a planar member machined from a substrate material;
 - b) an electrical conductor formed at least partly on the planar member; and
 - c) a gauge formed on the planar member and adapted to measure a physical effect on the planar member, the physical effect being indicative of a property of a fluid in contact with the planar member.
2. The sensor of claim 1, wherein the planar member includes a movable element.
3. The sensor of claim 2, wherein the electrical conductor forms a conducting coil on the movable element.
4. The sensor of claim 3, further comprising a source of current adapted to supply an electrical current to the conducting coil and a magnetic field source being adapted to generate a magnetic field, the magnetic field and the electrical current interacting to generate a force on the movable element that causes the movable element to vibrate.
5. The sensor of claim 4, wherein the magnetic field source comprises an electromagnet formed on the planar member.

6. The sensor of claim 4, wherein the movable element comprises a plate that is attached to the substrate along one side.

7. The sensor of claim 6, wherein the gauge comprises a strain gauge positioned near the side where the plate attaches to the substrate.

8. The sensor of claim 4, wherein the gauge comprises a differential pressure gauge arranged to measure the differential pressure of an acoustic wave generated in the fluid by the vibrating movable element.

9. The sensor of claim 8, further comprising an absolute pressure gauge and a temperature gauge.

10. The sensor of claim 4, wherein the movable element comprises a plate that is attached to the substrate by two arms, a first arm extending from one side of the plate and a second arm extending from an opposing side.

11. The sensor of claim 10, wherein the gauge comprises a strain gauge positioned at least partially on the first arm.

12. The sensor of claim 11, wherein the electrical conductor traverses the second arm.

13. The sensor of claim 3, wherein the movable element comprises a plate that is attached to the substrate by a first plurality of arms extending from one side of the plate and by a second plurality of arms extending from an opposing side.

14. The sensor of claim 13, wherein the gauge comprises a strain gauge positioned at least partially on one of the first plurality of arms.

15. The sensor of claim 1, wherein the electrical conductor couples with a Wheatstone bridge formed on the planar member.

16. The sensor of claim 1, wherein the electrical conductor couples with a series of capacitors formed on the planar member.

17. The sensor of claim 1, wherein the gauge comprises a thermometer and the electrical conductor couples with a heater formed on the planar member.

18. The sensor of claim 1, wherein the substrate material is selected from the group consisting of: monocrystalline silicon, poly-silicon, and silicon carbide.

19. A MEMS based fluid sensor comprising:
a planar member machined from a substrate material;
a movable plate machined from the planar member and remaining partially attached to the planar member;
an electrically conducting coil formed on a surface of the plate; and

23

at least one strain gauge positioned near where the movable plate remains attached to the planar member.

20. The sensor of claim 19, further comprising a magnetic field source adapted to generate a magnetic field approximately parallel or perpendicular to the surface of the movable plate.

21. The sensor of claim 19, wherein the movable plate comprises a flexural plate having four sides, the flexural plate being machined from the planar member along three sides and remaining attached to the planar member along one side.

22. The sensor of claim 19, wherein the movable plate comprises a plate attached to the planar member by at least two arms extending in opposite directions from the plate to the planar member.

23. A fluid sampling tool adapted to be moveable through a borehole that traverses an earth formation, comprising:

means for extracting a fluid from the earth formation into a container within the tool;

a Micro-Electro Mechanical Systems (MEMS) based sensor arranged to be in fluid contact with the fluid when held in the container; and

means for activating the MEMS based sensor to measure a physical property of the fluid under downhole conditions.

24. The tool of claim 23, wherein the MEMS based sensor comprises:
a planar member machined from a substrate material;
an electrical conductor formed at least partly on the planar member; and
a gauge coupled with the planar member and adapted to measure a physical effect on the planar member, the physical effect being indicative of a thermo-physical property of the fluid.

25. A sample bottle adapted to hold an oilfield reservoir fluid under downhole conditions comprising:
a container for holding the reservoir fluid; and
a Micro-Electro Mechanical Systems (MEMS) based sensor coupled to the container and arranged to be in fluid contact with the reservoir fluid when held in the container.

26. The sample bottle of claim 25, wherein the MEMS based sensor comprises:
a planar member machined from a substrate material;
an electrical conductor formed at least partly on the planar member; and
a gauge coupled with the planar member and adapted to measure a physical effect on the planar member, the physical effect being indicative of a property of the reservoir fluid.

27. The sample bottle of claim 25, further comprising a port adapted to receive the MEMS based sensor and through which the MEMS based sensor comes in contact with the reservoir fluid in the container.

28. A well adapted to produce a reservoir fluid from an underground reservoir to the surface comprising a Micro-Electro Mechanical Systems (MEMS) based fluid sensor adapted to measure a property of the reservoir fluid as it is being produced through the well.

29. A Micro-Electro Mechanical Systems (MEMS) based fluid sensor comprising:

a) a monolithic structure, machined from a substrate material, having a support portion allowing said monolithic structure to be attached to another structure, a plate portion capable of oscillating, and a flexible beam portion that decouples stress in said support portion from motion induced stress in said plate;

b) means for producing a magnetic field;

c) an electrical conductor, formed at least partly on said plate, allowing current flowing through said electrical conductor to interact with said magnetic field to induce oscillation of said plate;

d) a strain sensor, formed on said monolithic structure, adapted to detect movement of said plate; and

e) means for determining a thermodynamic property of a fluid in contact with said plate using said strain sensor detected plate movements.

30. The sensor of claim 29, wherein said electrical strain sensor is selected from the group comprising a Wheatstone bridge, a back-EMF sensor, and an electrical impedance sensor.

31. The sensor of claim 29, wherein said means for producing a magnetic field is a permanent magnet positioned adjacent to but not in contact with said plate.

1/7

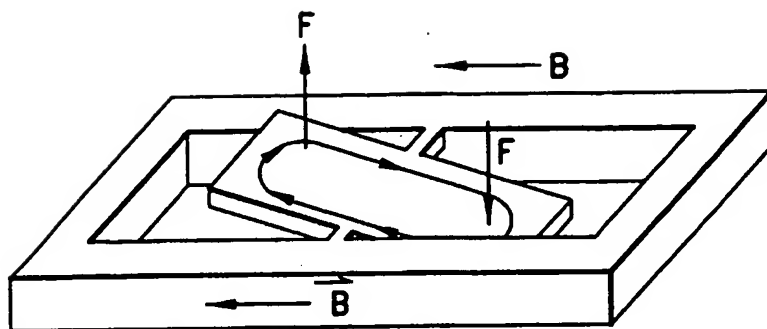


FIG. 1

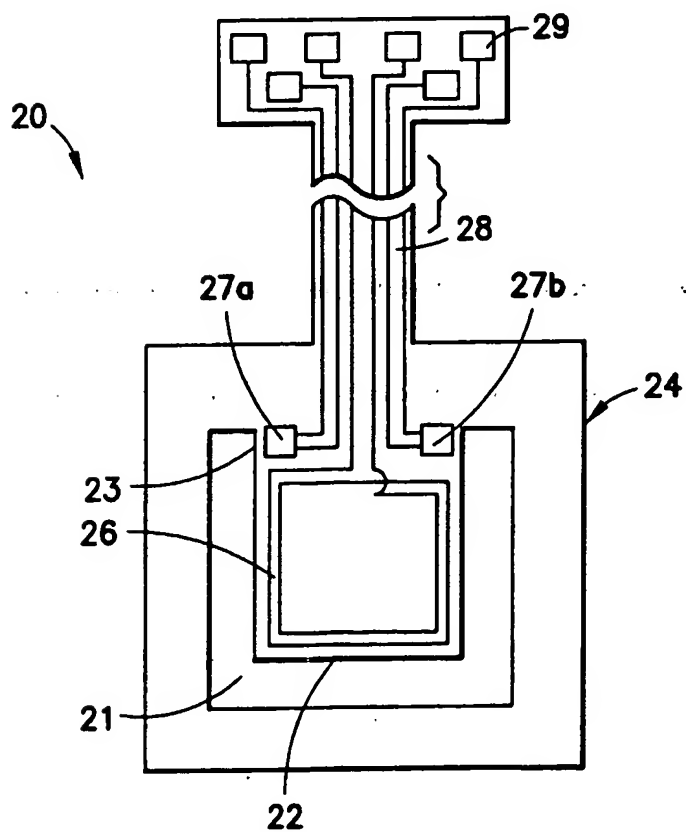


FIG. 2

2/7

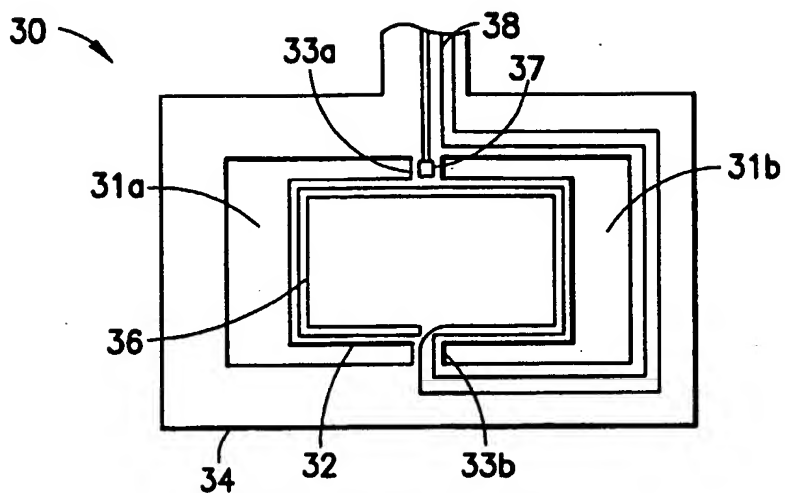


FIG. 3

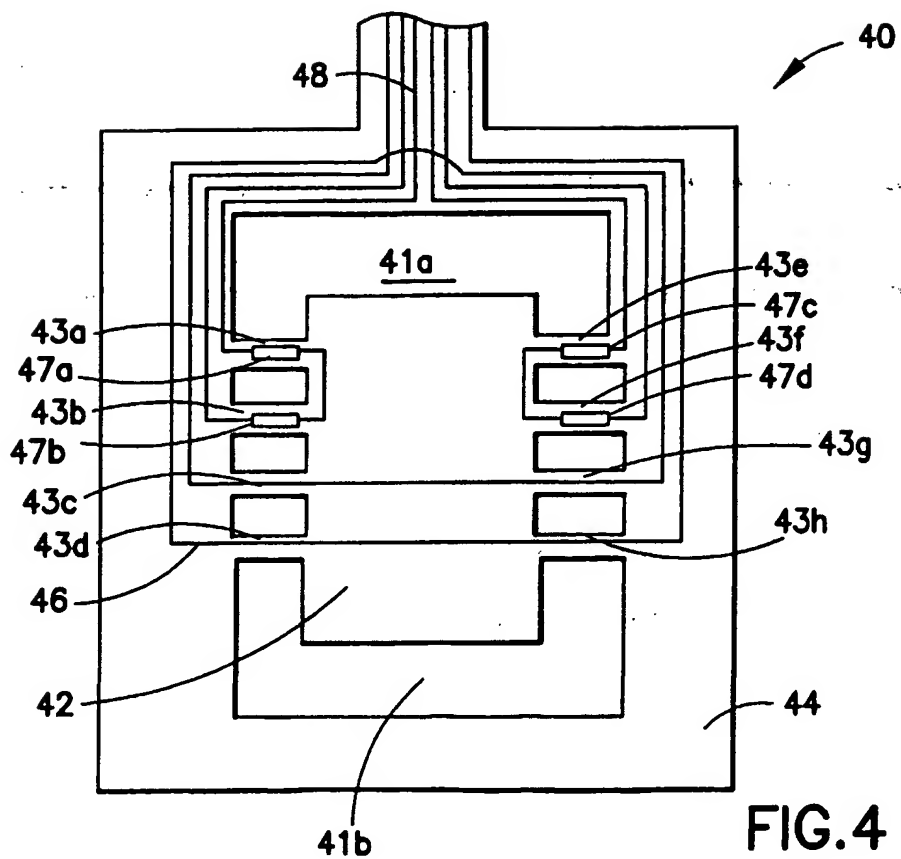


FIG. 4

3/7

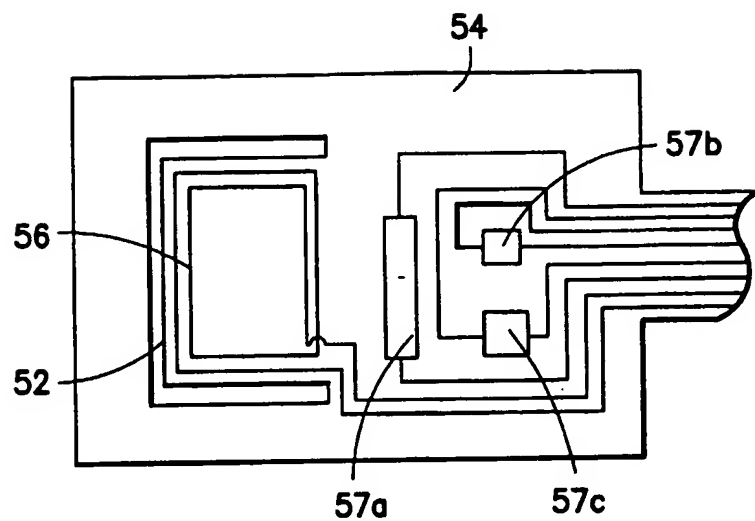


FIG. 5

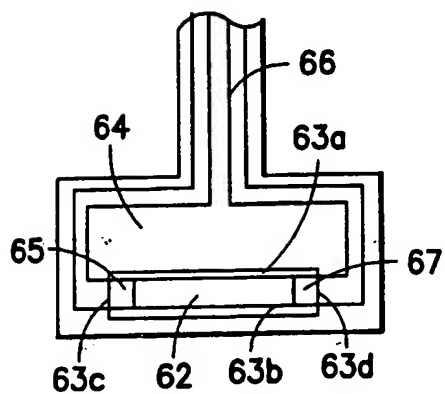


FIG. 6

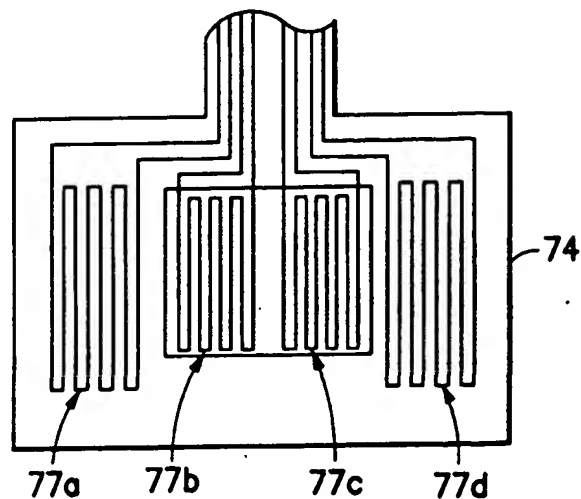


FIG. 7

4/7

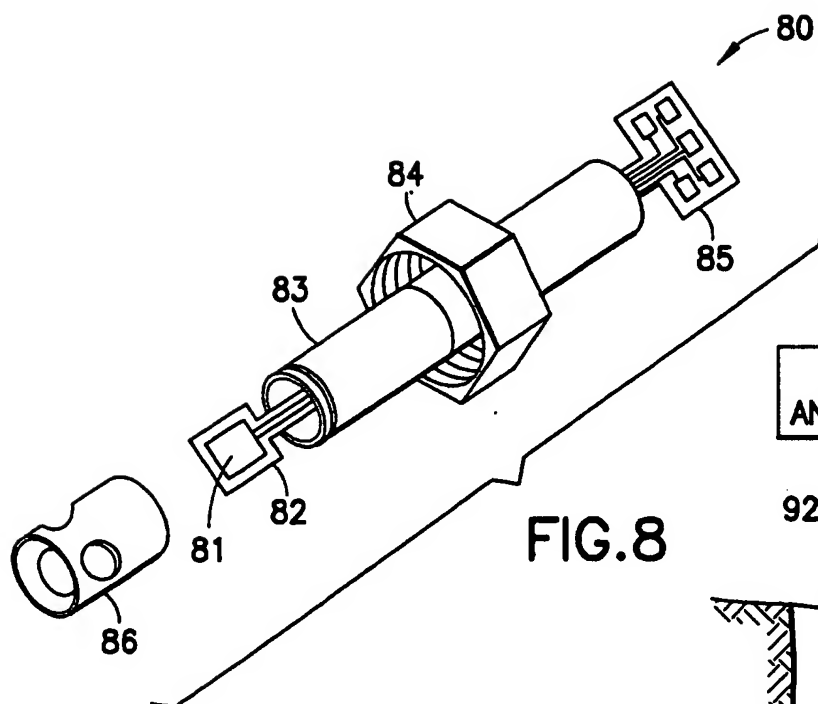


FIG. 8

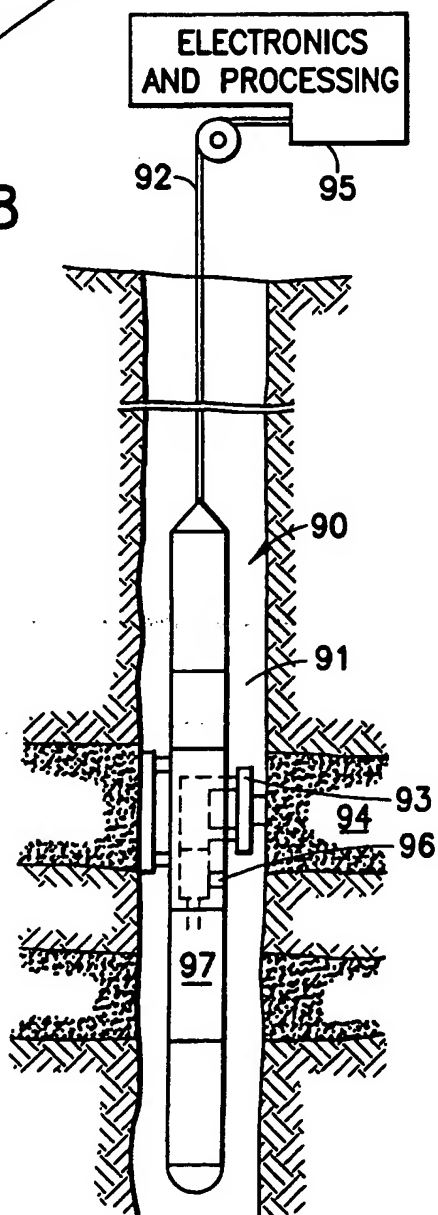


FIG. 9

5/7

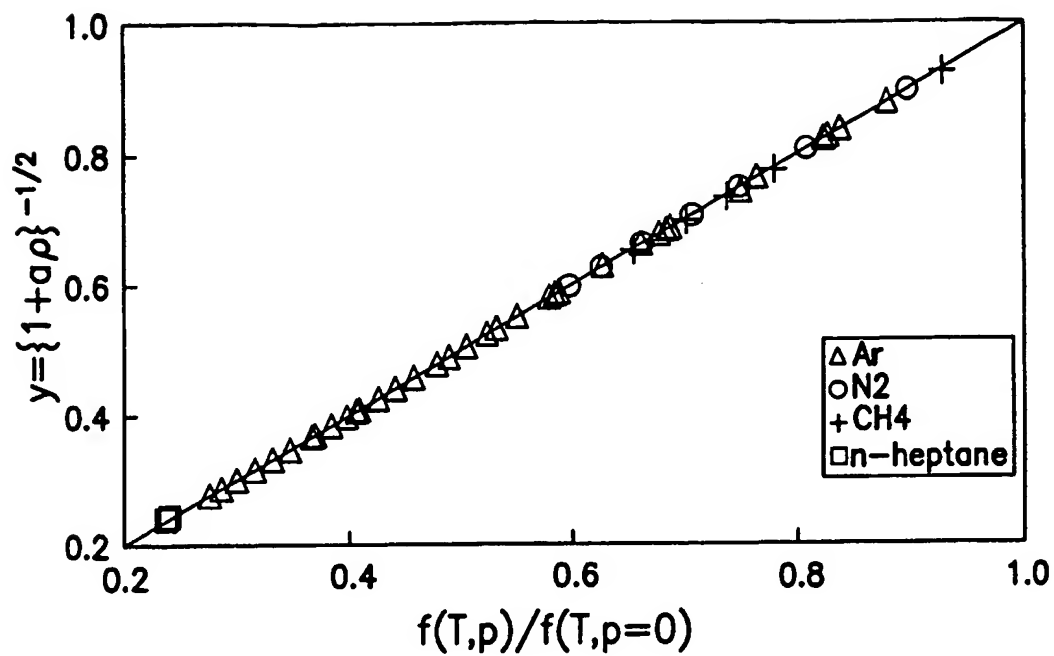


FIG. 10

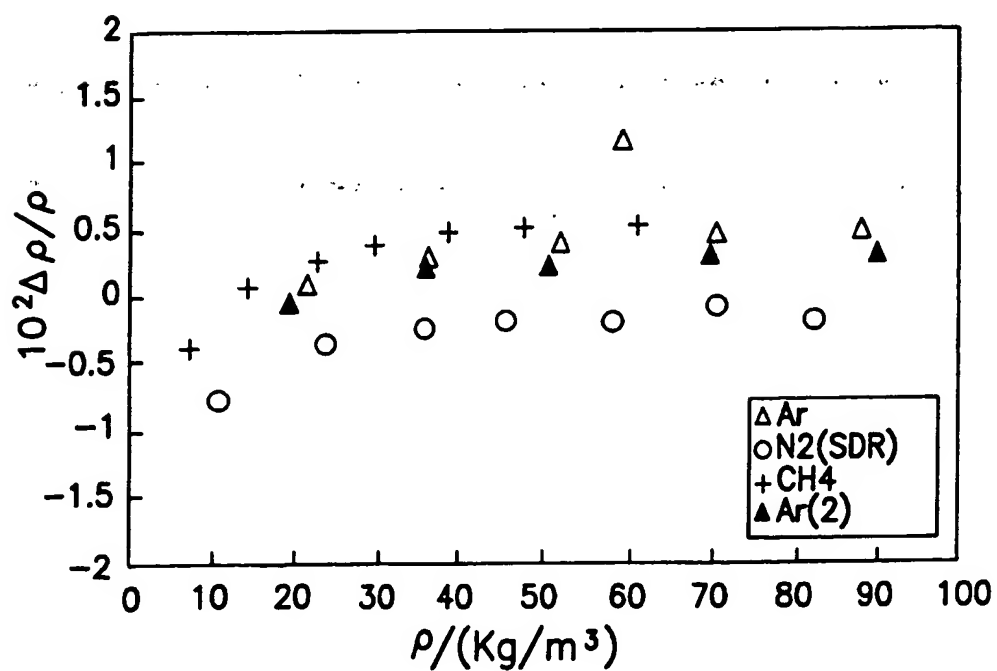


FIG. 11

6/7

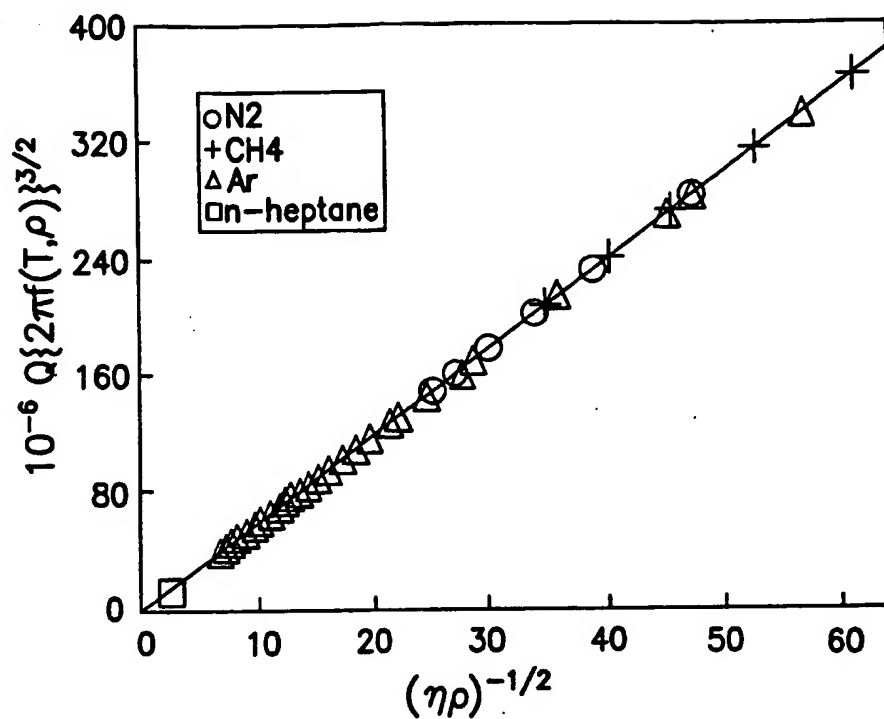


FIG.12

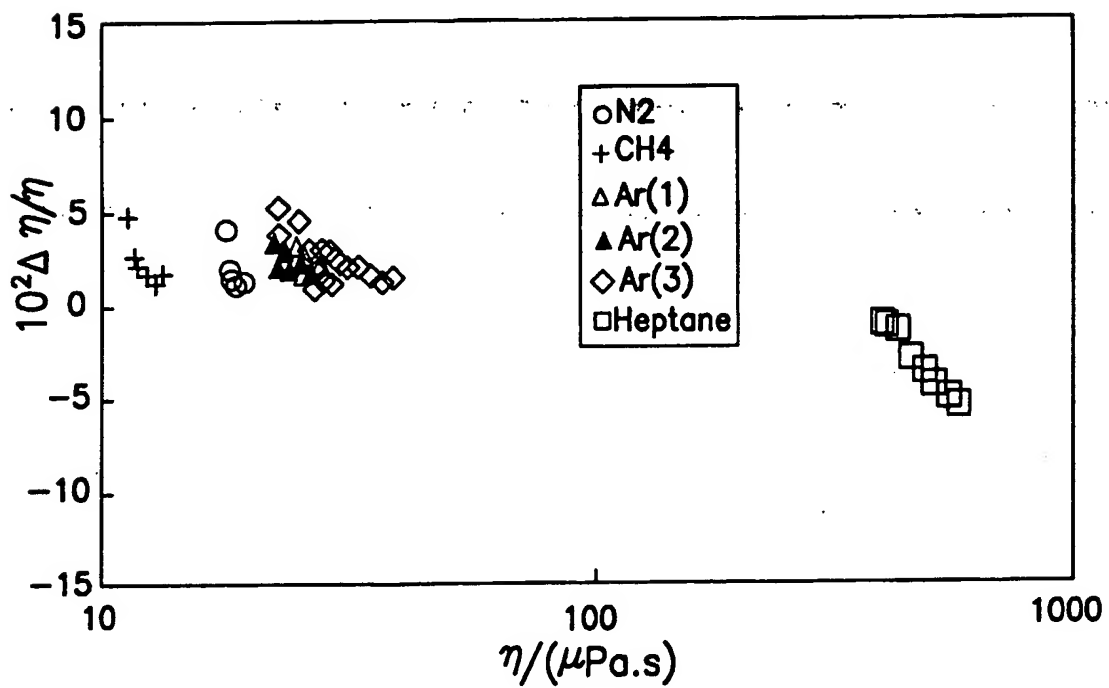


FIG.13

7/7

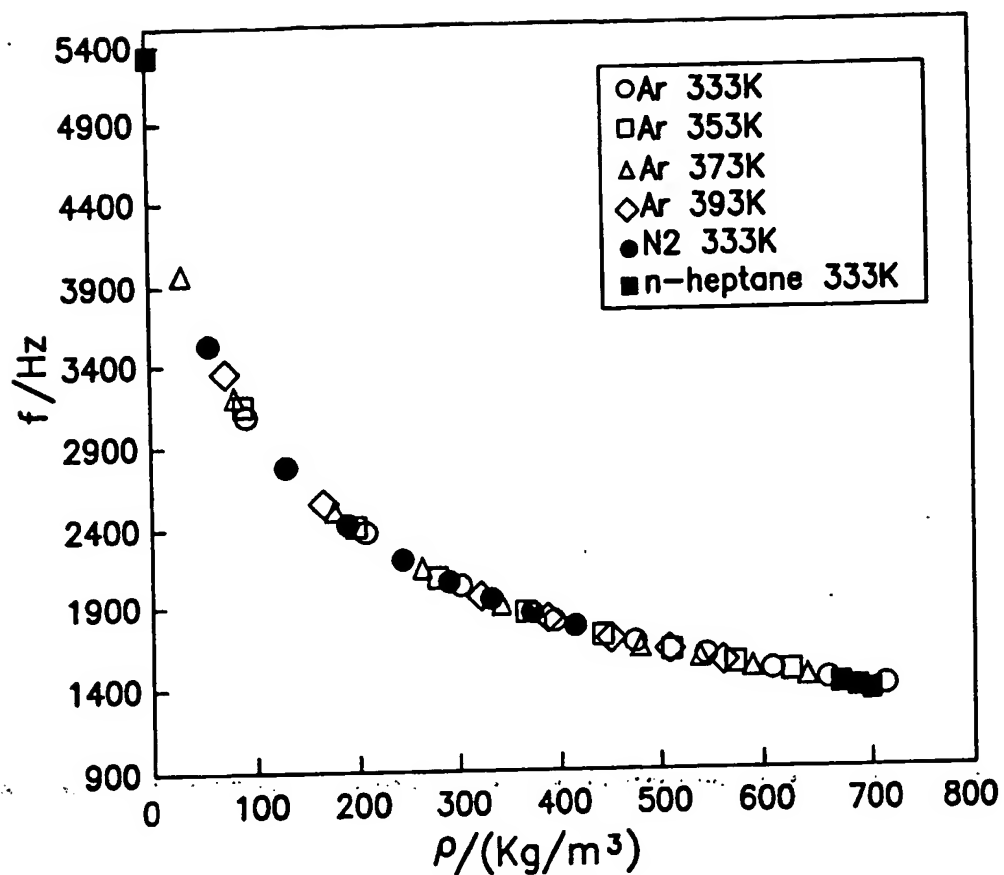


FIG.14

THIS PAGE BLANK (USPTO)

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ **BLACK BORDERS**
- ☐ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☐ **FADED TEXT OR DRAWING**
- ☒ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☐ **SKEWED/SLANTED IMAGES**
- ☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☐ **GRAY SCALE DOCUMENTS**
- ☐ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.

THIS PAGE BLANK (USPTO)